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ION BEAM TEXTURING

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ABSTRACT

Wehner and Hajicek have reported that a microscopic surface texture could be created by sputter etching a surface while simultaneously sputter depositing a lower sputter yield material onto the surface. A xenon ion beam source has been used to perform this texturing process on samples as large as three centimeters in diameter. Ion beam textured surface structures have been characterized with SEM photomicrographs for a large number of materials including - Cu, Al, Si, Ti, Ni, Fe, Stainless steel, Au, and Ag. Surfaces have been textured using a variety of low sputter yield materials - Ta, Mo, Nb, and Ti. The initial stages of the texture creation have been documented, and the technique of ion beam sputter removal of any remaining deposited material has been studied. A number of other texturing parameters have been studied such as the variation of the texture with ion beam power, surface temperature, and the rate of texture growth with sputter etching time.

INTRODUCTION

In 1942 Guenther Schulze and Tollmien¹ observed angular variations in the surface reflectivity of glow discharge cathodes that they hypothesized were the result of a submicroscopic surface texture. Later, Stewart and Thompson² published scanning electron photomicrographs of conical protrusions on tin and silicon surfaces that had been sputter etched with a radio-frequency argon ion source. They concurred with Guenther Schulze's postulation that the cones occur where low sputtering yield particles protect the underlying material from sputtering. Wehner and Hajicek³ have shown that surface texture can be created by supplying surfaces with lower sputtering yield atoms (see material) during sputter etching. It is thought that the low sputtering yield atoms agglomerate into microscopic regions which protect the higher sputtering yield atoms beneath. This paper presents experimental results obtained by ion beam texturing a number of elements and compounds with a xenon ion source. Scanning electron photomicrographs are shown that characterize the resulting surface texture in different regions of the periodic table. Photomicrographs are also presented which document the initial stages and growth of the surface texture. Variation of the texture with seeding material, ion energy, beam current and surface temperature have been studied.

ION BEAM TEXTURING PROCESS

An electron bombardment xenon ion source was used.⁴ This source has evolved from an auxiliary propulsion thruster which is intended for station keeping spacecraft. The ion beam is accelerated and

focused by a multiaperture double electrode system. The source is capable of operating at net accelerating potentials between 500 and 2000 volts. The beam current is adjustable between 10 milliamperes and 200 milliamperes. At a net accelerating potential of 1000 volts and a beam current of 100 ma the current density at the center of the beam, 10 centimeters from the source is 2.0 ma/cm^2 . The specific operating conditions were varied because of the variation in the melting point of the target material.

The material to be textured was located 10 centimeters from the source and centered in the ion beam. It was mounted such that the source axis was normal to its surface. The seed material was mounted on a separate support, it was oriented at a 45° angle with respect to source axis. It was located within the beam envelope very close to but not touching the target. The ion beam simultaneously sputtered both the target and the seed material. Sufficient seed material was sputter deposited onto the target to create a dense uniform texture over areas up to 3 centimeters in diameter.

MORPHOLOGY OF TEXTURED ELEMENTS

Ion beam texturing using tantalum as the seed material was attempted on thirty different elements. Those elements (twenty-six in number) that were successfully textured are shaded in figure 1. The texturing process was unsuccessful for the four crosshatched elements in the periodic chart. All of the elements that could not be textured are in columns VB and VIB of the periodic chart. It is interesting to compare this observation with the periodicity effect of the

low energy sputter yield that has been reported by Wehner⁵ and by Wehner and Rosenberg.⁶ The elements (Nb, Mo, Ta, W) that showed no signs of texture are all relatively low sputter yield materials. However, several elements (C, Si, Ti, and Zr) have lower sputter yields than tantalum, but nevertheless were still textured. Conversely, tungsten, which has a higher sputter yield than tantalum, did not texture. The elements in column IVB of the periodic chart all could be textured; however, zirconium textured at a faster rate (4.3×10^{-6} cm/min) than either hafnium (6.7×10^{-7} cm/min) or titanium (1.0×10^{-6} cm/min).

It is extremely difficult to describe the morphological (form and structure of a surface) surface differences that occurred on the twenty-six elements that were textured. A large number of elements had textures that can be classified as either a ridge structure or a cone structures. Figure 2a is a photomicrograph of the convoluted ridge morphology that occurred on nickel and many of the other low sputtering yield materials (Ti, Fe, Co, Zr, Hf, Gd). Many of the higher sputter yield materials had morphologies that look like densely packed cones or needles. The scanning electron photomicrograph of copper shown in figure 2b, is an example of this second kind of morphology. The other elements that exhibit this type of structure include Mg, Cr, Ag, Au, Hg, Al, C, Si, Se, Pb, and Bi. The remaining materials that textured but are neither ridge structure or cone structure are Cd, Be, Zn, In, Sn, and Sb. Their surface textures were all unique. Analysis of the X-ray energy dis-

persion of textured silicon confirmed Wehner's postulation that the regions on the top of cones had considerably more tantalum than the regions between the cones.

In addition to the elements a few compounds, alloys, and polymers such as stainless steel, CrAu, and teflon have been textured. The resulting surface morphologies can also be classified into ridge and cone categories.

THE EVOLUTION OF SURFACE TEXTURE

A series of copper samples were ion beam textured at a net accelerating voltage of 1000 volts and a beam current of 100 ma for periods of 2, 4, 8, 16, and 32 minutes. The resulting surface textures were then examined with a scanning electron microscope. Figure 3 shows two views of the 2 minute textured surface. The vertical view shows the distribution of the tantalum nucleation sites. They seem to be composed of islands 0.1 to 0.5 μm in diameter surrounded by a thinner more uniformly distributed component. The angular view shows the depth of the structure and the initiation of the texture. Figure 4 shows the surface texture after 4 minutes of ion beam texturing. The peaks are on the average somewhat larger in diameter than after 2 minutes and in a few locations appear to be lined up in rows. Clearly after 4 minutes the cone structure is well underway. After 8 minutes the surface texture looked almost exactly like figure 2b. Further exposure to the ion beam only results in increased depth of the structure. Note that some of the cones appear to be hollow.

Measurements of the average copper cone height as a function of the ion beam exposure time are graphed in figure 5. The curve has a change in slope between the 4 minute point and the 8 minute point. This is probably a result of the transition from the initial stages of texturing to the lower sputtering yield later stages where more sputtered atoms are trapped by the cones. The cone growth rate in the later state is estimated at 0.88×10^{-5} cm/min. At the same beam conditions the copper etch rate with no tantalum present was 1.2×10^{-5} cm/min. By way of comparison stainless steel, a lower sputtering yield material, has a texture growth rate of 1.0×10^{-6} cm/min under the same conditions.

PARAMETERS OF TEXTURING

There are several variables in the texturing process such as the kind of seed material, its' arrival rate at the target, the net accelerating voltage, the beam current, and the target temperature. A complete analysis of the large number of parametric perturbations is beyond the scope this paper, nevertheless some specific observations may be of interest.

Figure 6 shows textured copper surfaces that resulted in (a) tungsten seed material and (b) with Al_2O_3 seed material. The morphologies were different from each other and different from those created with a tantalum seed material (fig. 2b). Different seed materials do not always result in a changed morphology. When silicon was textured with tantalum, molybdenum, or titanium the resulting morphologies were found to be nearly identical.

The density of the texture (the number of cones or ridges per unit area) was found to be dependent on the arrival rate of the seed material. At low arrival rates the cones were more widely separated and more perfectly formed. It is also possible for the seed material arrival rate to be so high that no texture forms on the target.

Preliminary measurements of the growth rate of stainless steel texture found it to be proportional to both beam current and net accelerating potential.

Surface temperature is also very important to the texturing phenomena. For example, if the normal texturing process of copper with tantalum seed material was interrupted after every minute of operation and allowed to cool for several minutes, no texture was created. Instead the copper became coated with tantalum. This may result because of the temperature activated surface mobility of the seed material postulated by Wehner.

CONCLUDING REMARKS

An ion beam source was found to be a controlled method for texturing surfaces. Twenty-six elements have been textured and the resulting surface morphologies have been characterized. A large number of elements had textures that can be classified as either a ridge structure or a cone structure. Scanning electron photomicrographs of the nucleation of the seed material and the growth of copper cones support Wehner's postulation of the texturing mechanism. Measurements of cone growth with texturing time showed that the growth rate decreased after the initial stages of texturing. Preliminary observa-

tions on the interrelationships between materials and the texturing parameters suggest the complexity of the process and the need for future experiments and analysis.

The surface morphology created by ion beam texturing has several exciting potential applications. This type of surface treatment can be used to modify reflectance and emissivity, to decrease secondary electron emission, to promote thin film adhesion, to increase catalytic reactions, and to reduce the effective sputtering yield. The reflectance of silicon single crystals has been reduced to a few percent over the solar spectrum by ion beam texturing the surface. Preliminary results have also shown that the secondary electron emission yield of copper and titanium can be reduced by ion beam texturing.

REFERENCES

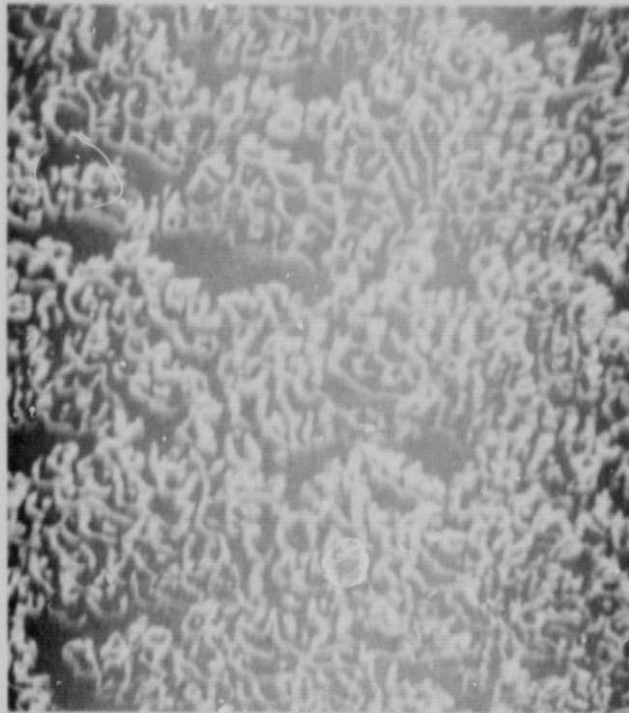
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2. A. D. G. Stewart and M. W. Thompson, J. Material Sci. 4, 56 (1969).
3. G. K. Weher and D. J. Hajick, J. Appl. Phys. 42, 1145 (1971).
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6. G. K. Wehner and D. Rosenberg, J. Appl. Phys. 32, 887 (1961).

PERIODIC CHART OF THE ELEMENTS

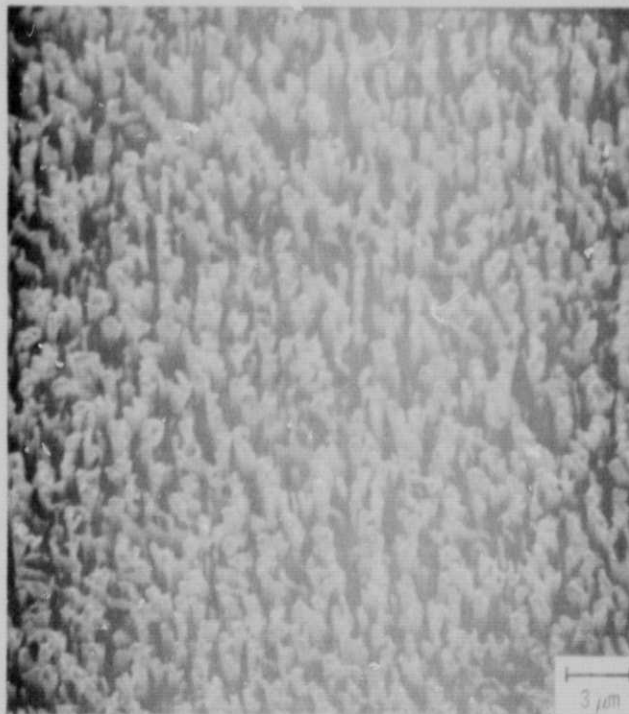
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1 H																		1 H	2 He
3 Li	4 Be												5 B	6 C	7 N	8 O	9 F	10 Ne	
11 Na	12 Mg												13 Al	14 Si	15 P	16 S	17 Cl	18 Ar	
19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr		
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe		
55 Cs	56 Ba	57 La	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn		
87 Fr	88 Ra	89 Ac																	
* LANTHANUM SERIES																			
			58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu			
† ACTINIUM SERIES																			
			90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md					

Figure 1. - The periodic chart of the elements. The shaded elements were successfully textured using tantalum as the seed material. The texturing process was unsuccessful for the cross-hatched materials.

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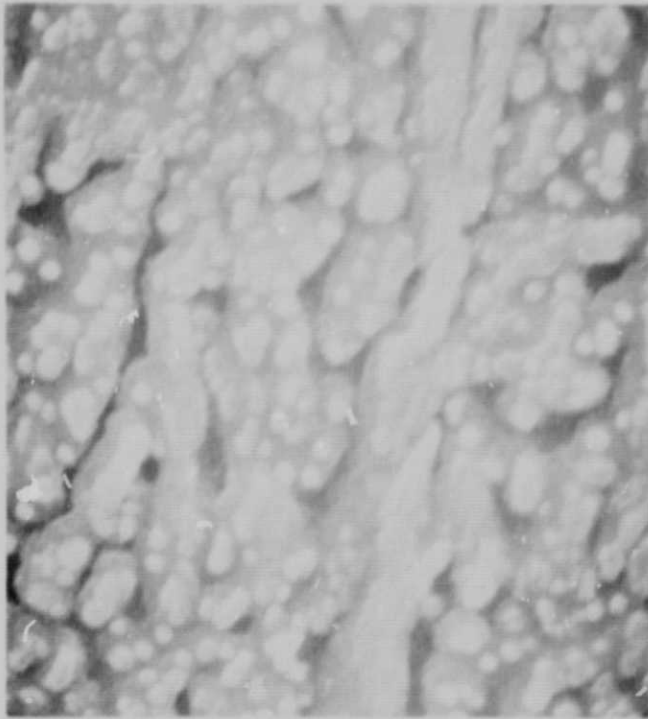


(a) Nickel.



(b) Copper.

Figure 2 - Scanning electron photomicrographs of ion beam textured nickel (a) and copper (b), tantalum was the seed material. The net accelerating potential of the ions was 1000 volts and beam current was 100ma. 3000X, 40° tilt.



(a) 0° tilt.



(b) 40° tilt.

Figure 3. - Scanning electron photomicrographs of an ion beam textured copper surface (two minute exposure), tantalum was the seed material. The net accelerating potential was 1000 volts and the beam current was 100ma. 10,000X.



(a) 0° tilt.



(b) 40° tilt.

Figure 4. - Scanning electron photomicrographs of an ion beam textured copper surface (four minute exposure), tantalum was the seed material. The net accelerating potential was 1000 volts and the beam current was 100ma. 10,000X.

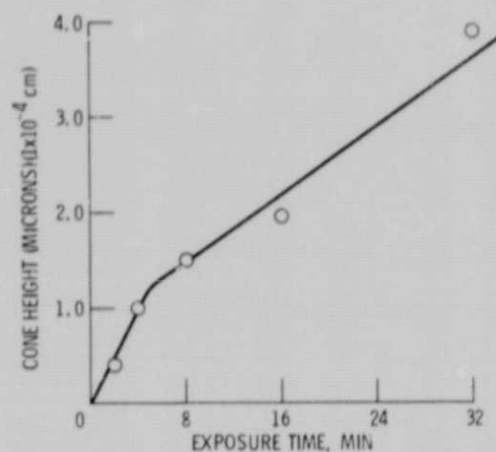
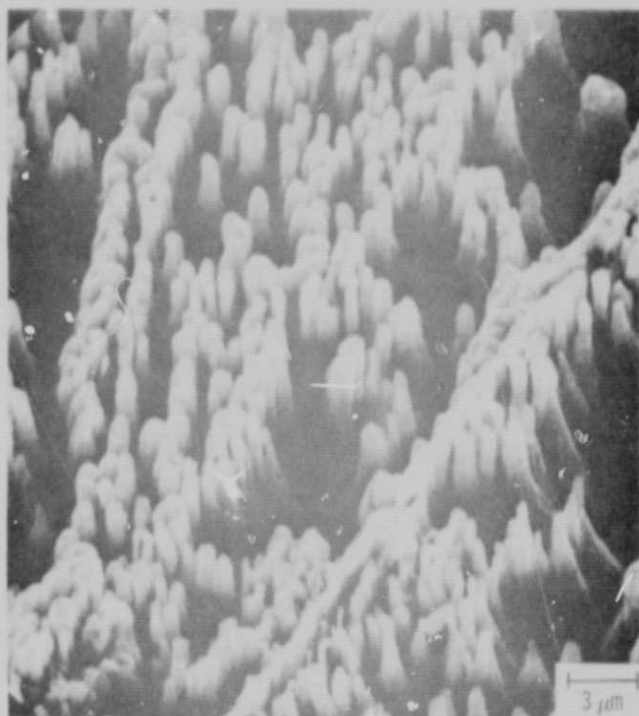


Figure 5. - Measurements of the copper cone height with respect to ion beam exposure time. The Xexon ion beam operated at 1000 volts and 100 ma. Tantalum was the seed material.



(a) 3000X 40° tilt.



(b) 1000X 40° tilt.

Figure 6. - Scanning electron photomicrographs of ion beam textured copper, in 11a tungsten was the seed material and in 11b, Al_2O_3 was the seed material. The net accelerating potential was 1000 volts and the beam current was 100ma.